

March 8, 1977

A STUDY OF THE DECAY RATE OF DEF AND FOLEX
AS A FOLIAR SPRAY ON COTTON IN
KERN COUNTY, CALIFORNIA
SEPTEMBER 1975

By

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INTRODUCTION

DEF (S,S,S-Tributyl phosphorotrithioate) is registered for use in California as a cotton defoliant. Its acute oral LD₅₀ (rat) is 200 mg/kg, while its dermal LD₅₀ is greater than 1000 mg/kg. Label recommendations are for 1-1/3 to 2 pints (1 to 1-1/2 lbs. actual) per acre for complete defoliation; and 1 to 1-1/2 pints (3/4 to 1-1/8 lbs. actual) per acre for bottom defoliation; 3 pints (2-1/4 lbs. actual) per acre for complete defoliation of rank cotton. Applications should not be made within 7 days of harvest.

Folex (Tributyl phosphorotrithioite) is also registered for use as a cotton defoliant. Technical Folex has an acute oral LD₅₀ of 1653 mg/kg. The dermal LD₅₀ is 400 mg/kg. The label recommends using 1/2 pint (3/8 lbs. actual) per acre for pre-conditioning; 1-1/3 pints (1 lbs. actual) per acre for bottom defoliation; and 1-1/2 to 2 pints (7/8 to 1-1/2 lbs. actual) per acre for total defoliation. Both DEF and Folex cause leaves to drop in green condition.

APPLICATION

As part of a study of potential safety hazards, an analysis was made of the rate of decay of DEF and Folex applied as a cotton defoliant. During the same season, residue decay of DEF on foliage as well as release of residues into the air of several fields in nearby Tulare County. As these pesticides decay, butyl mercaptans present in their formulations are released to the air and are considered by most persons to have a foul odor. When this odor is intense in nearby residential areas or schools, it tends to result in discomfort of varying numbers of persons who experience nausea, vomiting, headaches and dizziness.

The defoliants studied had been applied to five fields in Kern County, California.

The five fields were treated as follows:

Field 1: (70 acres)

September 23, 1975
1.5 pt. DEF (1-1/8 lbs. actual)
1.5 pt. Bolls Eye
.83 qt. Nutra Wet
12 gal. water

Field 2: (100 acres)

September 24, 1975
1 qt. Folex (1-1/2 lbs. actual)
1.5 qt. Bolls Eye
10 gal. water

Field 3: (96 acres)

September 30, 1975
1.5 pt. DEF (1-1/8 lbs. actual)
0.5 pt. Paraquat
0.1 qt. Kerntox B-99 (spreader)
10 gal. water

Field 5: (60 acres)

October 8, 1975
3 pt. DEF (2-1/4 lbs. actual)
0.5 pt. Accelerate (endothall)
0.1 qt. spreader

Field 5 was treated by a ground rig, while the other 4 fields were treated aerially.

Field 4: (approx. 26 acres)

October 1, 1975
1 qt. Folex (1-1/2 lbs. actual)
0.5 qt. Paraquat
0.1 pt. K-90
10 gal. water

SAMPLING

Sampling was done by taking triplicate samples of 100 leaf punches, 2.5 cm in diameter, on a diagonal across the field. Duplicate samples were taken for surface and penetrated analysis, while the third was taken for total residue. Results for the first two were averaged before being plotted on the graphs.

ANALYTICAL PROCEDURES

Extraction

The procedure used for the extraction of dislodgeable, penetrated, and total residues from leaf punches was originally published by Gunther in "The Bulletin of Environmental Contamination and Toxicology", 9, 243-249, 1973. It has been documented several times in detail, with modifications that were made to accommodate the various pesticides and their metabolites. The sample container and leaf punches are weighed and the gross weight recorded.

Total Residues

1. The leaf punches are transferred to a blending jar. The empty sample container is again weighed and the net weight of the punches recorded.
2. Approximately 50 gm of sodium sulfate and 100 ml of ethyl acetate are added.
3. The sample is blended at high speed for 3 minutes, keeping the blender cup cool by immersing it in a container of cool water. The blender cup is removed and the sample allowed to settle.
4. An aliquot is decanted into a Teflon-capped bottle and may be stored in the freezer prior to clean up and analysis.

Dislodgeable Residues

1. Fifty ml of water and approximately 4 drops of Sur-Ten solution (1:50) are added to the sample containers. The containers are capped and placed in a multi-purpose rotator and rotated at 30 cycles/min. for 60 min. The aqueous solution is decanted through a glass wool plug into a 500 ml separatory funnel.
2. The punches are rotated a second time, using 50 ml of water and 4 drops of Sur-Ten solution for 30 min. This is added to the first extraction.
3. The sample is then hand-shaken for approximately 10 seconds with 30 ml of water. The container is drained into the separatory funnel with the first two extractions.
4. The aqueous solution is extracted three times with 50 ml of ethyl acetate. The solvent is filtered through sodium sulfate into a glass stoppered mixing cylinder and the volume is recorded. The solvent is mixed in the cylinder. An aliquot is decanted into a Telfon-capped bottle and stored in the freezer prior to cleanup and analysis.

Penetrated Residues

1. After the last water rinse is drained for the dislodgeable residue, the punches are transferred to a blender jar. The empty sample container is weighed and the net weight of the punches recorded.
2. Approximately 50 gm of sodium sulfate and 100 ml of ethyl acetate are added.
3. The sample is blended and handled the same as the total residue sample.

Gas Chromatography

Instrument: Varian 2700, FPD Detector

Column: 3' x 2 mm I.D. of 3% OV-101 on 100/120 mesh Gas Chrom Q operated at 200°C

Carrier Gas: N₂ @ 30 cc/min

Retention Time: 3 minutes

DEF and Folex residues are examined in the same manner since DEF is the oxidized analog of Folex.

RESULTS

Weather observations for the study period can be found in Table 1. The average minimum and maximum temperatures were 64.2°F and 84.2°F respectively. The temperature varied greatly between the time when fields 1 and 5 were studied.

Results of the analysis can be seen in Tables 2-6 and the graphs that follow. As can be seen, degradation rates were similar for fields 1 through 4 where 1.5 to 2 pints per acre were applied aerially. Field 5 was treated by ground rig with 3 pints per acre and shows a slower more erratic rate of decline.

TABLE 1: DAILY TEMPERATURE AND PRECIPITATION

Weather observations were taken in Bakersfield, Kern County, California

DATE (1975)	TEMPERATURE (°F)		PRECIPITATION (IN.)	FIELD NUMBERS
	MINIMUM	MAXIMUM		
9-23	70	97		1
9-24	70	100		2
9-25	69	103		
9-26	69	100		
9-27	71	97		
9-28	66	91		
9-29	66	91		2
9-30	67	94		3
10-1	68	95		4
10-2	67	93		1
10-3	67	93		3
10-4	69	93		4
10-5	62	98		
10-6	64	92	0.22	
10-7	53	68		
10-8	50	75		5
10-9	54	79		
10-10	57	79	0.09	
10-11	<u>61</u>	<u>71</u>	0.07	5
Average	64.2	84.2		

TABLE 2: DEF RESIDUES IN FIELD 1 FOLLOWING APPLICATION TO COTTON

SAMPLE	HOURS POST-APPLICATION	DEF RESIDUES (PPM)		
		SURFACE	PENETRATED	TOTAL
TR-1	1	81.2	219	161
TR-2	1	60.2	182	
TR-3	1			
TR-4	6	57.5	105	145
TR-5	6	70.1	95.5	
TR-6	6			
TR-7	21	28.0	132	100
TR-8	21	19.5	91.1	
TR-9	21			
TR-10	29	12.3	85.4	79.6
TR-11	29	8.74	60.9	
TR-12	29			
TR-13	46	4.63	49.6	48.5
TR-14	46	4.63	37.8	
TR-15	46			
TR-16	69	4.14	42.7	47.3
TR-17	69	4.83	38.6	
TR-18	69			
TR-19	145	1.96	16.2	16.6
TR-20	145	1.58	13.4	
TR-21	145			
TR-22	216	1.00	10.4	14.9
TR-23	216	0.24	10.8	
TR-24	216			

TABLE 3: FOLEX RESIDUES IN FIELD 2 FOLLOWING APPLICATION TO COTTON

SAMPLE	HOURS POST-APPLICATION	FOLEX RESIDUES (PPM)		
		SURFACE	PENETRATED	TOTAL
W-1	1	62.0	119	173
W-2	1	66.5	188	
W-3	1			
W-4	24	57.1	121	142
W-5	24	43.7	91.6	
W-6	24			
W-7	46	19.4	68.8	58.2
W-8	46	16.0	66.5	
W-9	46			
W-10	125	2.03	13.6	11.0
W-11	125	2.02	14.3	
W-12	125			

TABLE 4: DEF RESIDUES IN FIELD 3 FOLLOWING APPLICATION TO COTTON

SAMPLE	HOURS POST-APPLICATION	DEF RESIDUES (PPM)		TOTAL
		SURFACE	PENETRATED	
PR-1	1	105	116	
PR-2	1	115	102	
PR-3	1			214
PR-4	6	64.9	118	
PR-5	6	54.7	135	
PR-6	6			142
PR-7	26	36.0	109	
PR-8	26	40.0	121	
PR-9	26			126
PR-10	53	12.5	86.0	
PR-11	53	18.3	115	
PR-12	53			85.5
PR-13	73	1.3	22.7	
PR-14	73	2.8	29.5	23.8
PR-15	73			

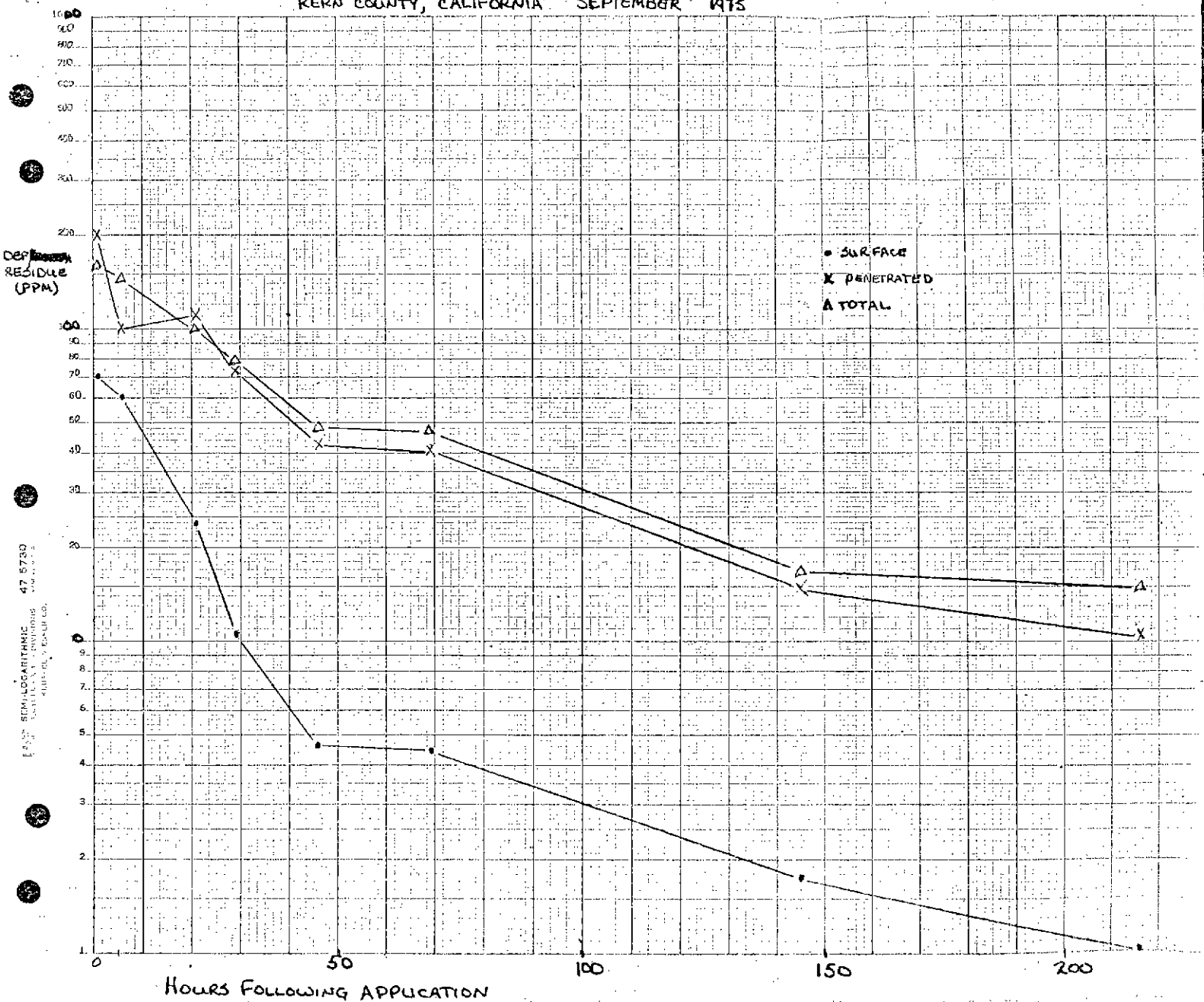
TABLE 5: FOLEX RESIDUES IN FIELD 4 FOLLOWING APPLICATION TO COTTON

SAMPLE	HOURS POST-APPLICATION	FOLEX RESIDUES (PPM)		TOTAL
		SURFACE	PENETRATED	
AR-1	1	98.5	102.6	
AR-2	1	136.9	125.6	
AR-3	1			189
AR-4	6	46.0	122	
AR-5	6	49.4	148	
AR-6	6			168
AR-7	25	23.2	84.0	
AR-8	25	24.6	122	
AR-9	25			175
AR-10	48	3.1	41.5	
AR-11	48	5.9	65.9	
AR-12	48			64.2

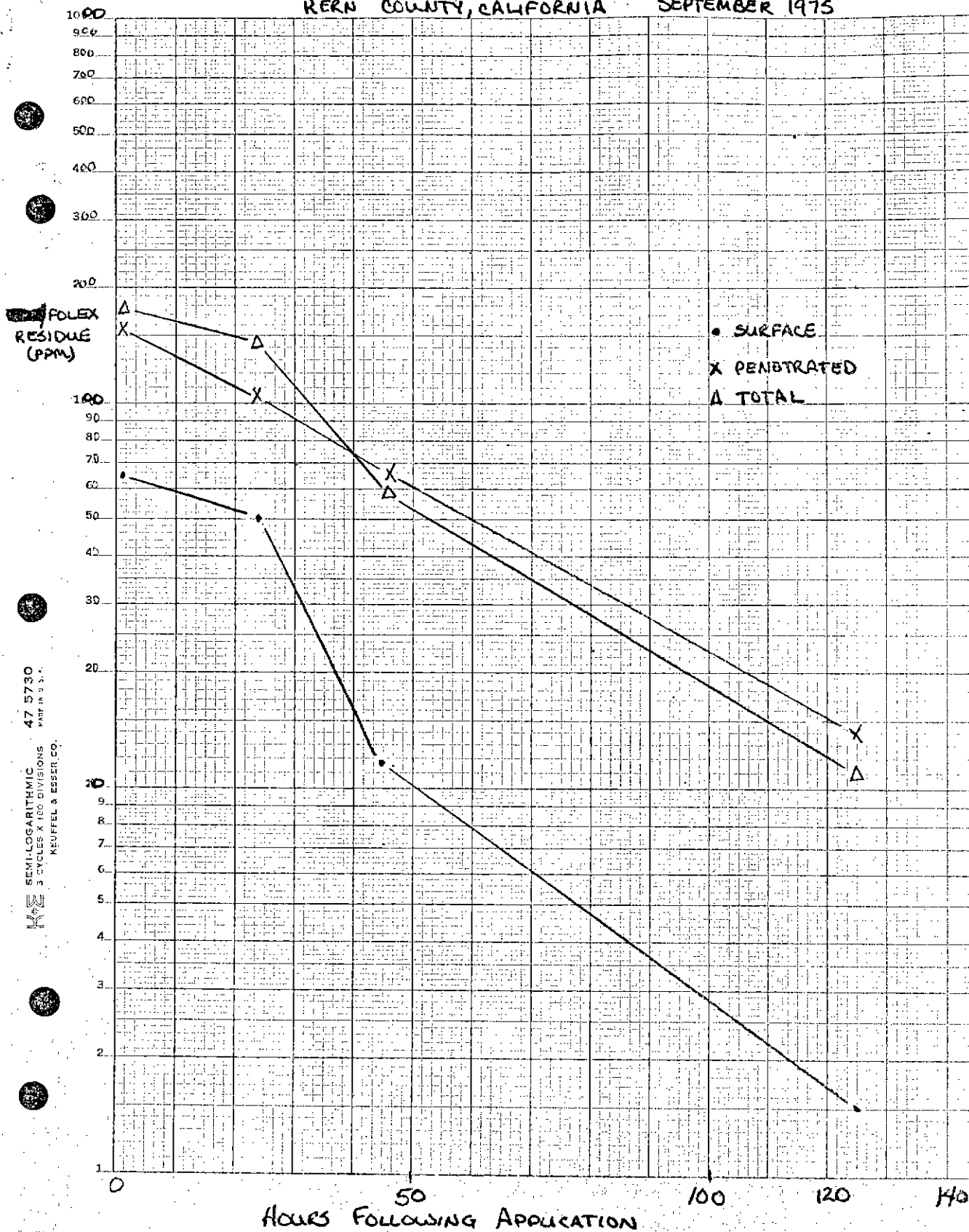
TABLE 6: DEF RESIDUES IN FIELD 5 FOLLOWING APPLICATION TO COTTON

SAMPLE	HOURS POST-APPLICATION	DEF RESIDUES (PPM)		TOTAL
		SURFACE	PENETRATED	
1	Before Application	-	-	
2	Before Application	-	-	
3	Before Application			<1.0
4	1	186	262	
5	1	130	168	
6	1			286
10	6	88.6	164	
11	6	86.0	147	
12	6			283
13	22	105	184	
14	22	146	162	
15	22			261
16	28	64.5	172	
17	28	57.4	167	
18	28			212
Soil	28			34.5
19	45	50.5	187	
20	45	54.6	171	
21	45			178
22	51	38.1	167	
23	51	33.1	155	
24	51			185
25	69	19.5	162	
26	69	16.7	169	
27	69			155

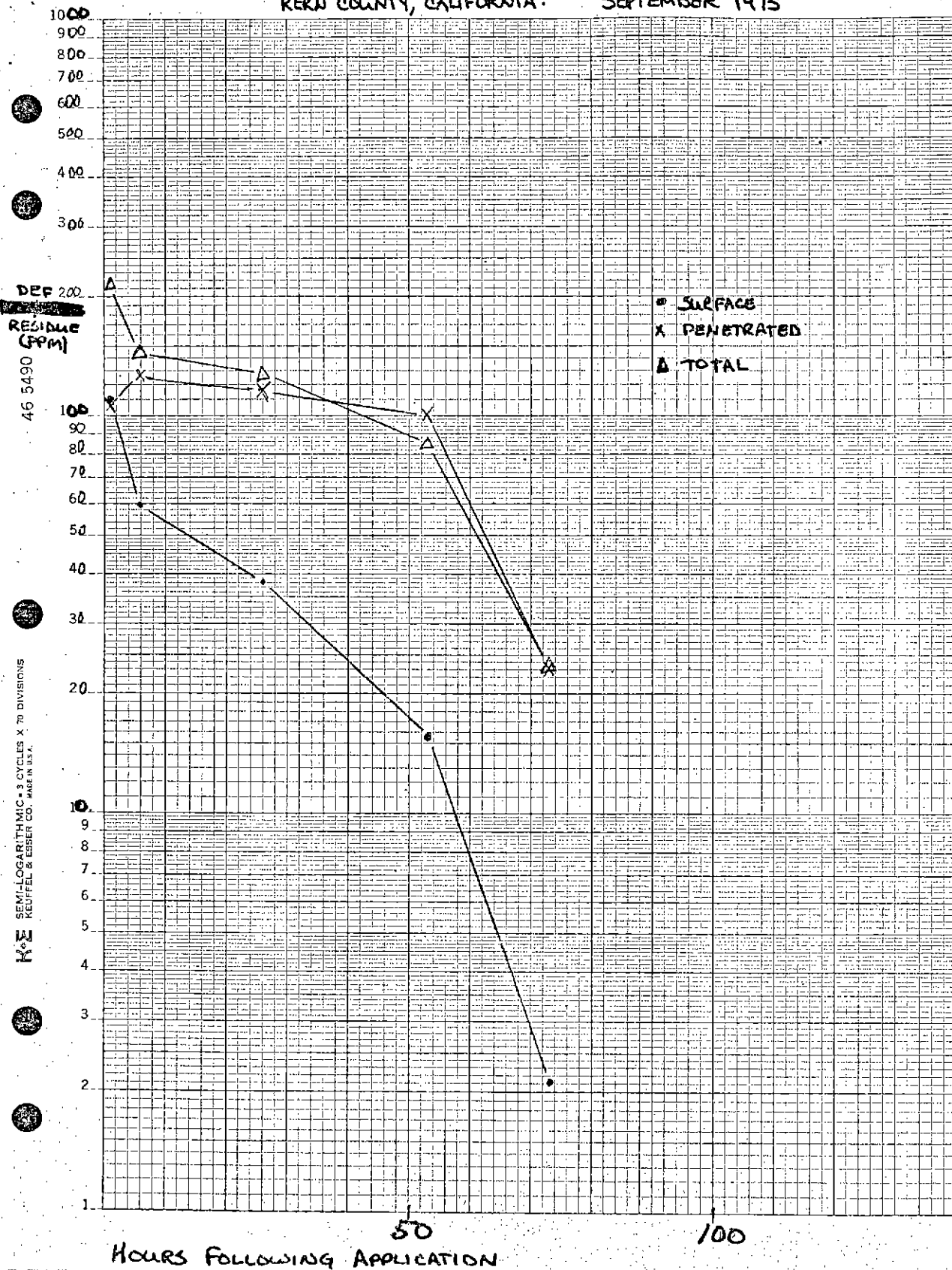
GRAPH 1: ~~DEF~~ RESIDUE ON COTTON IN FIELD 1 (PPM)
KERN COUNTY, CALIFORNIA SEPTEMBER 1975



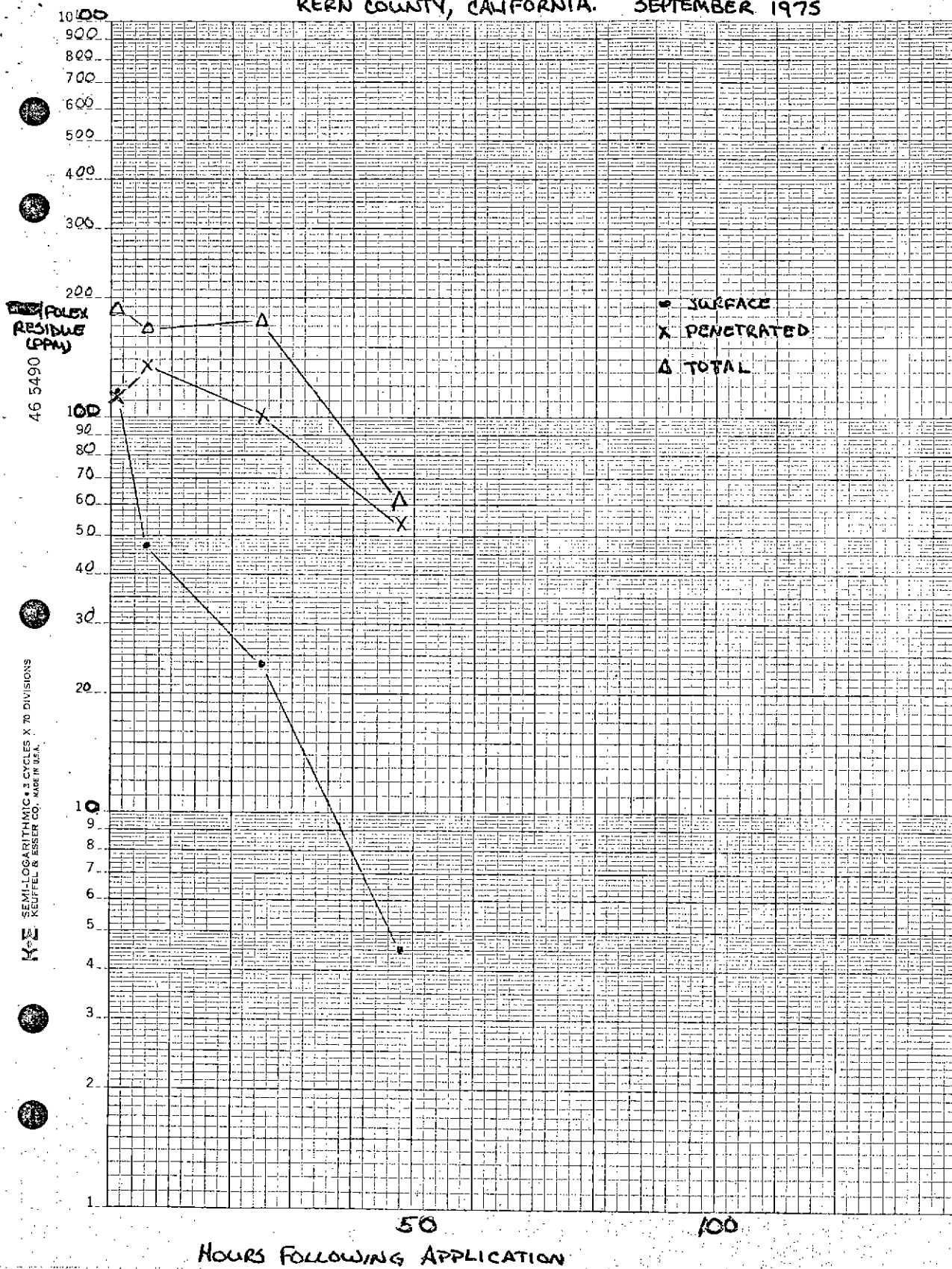
GRAPH 2: ~~FOLEX~~ FOLEX RESIDUES ON COTTON IN FIELD 2 (PPM)
KERN COUNTY, CALIFORNIA SEPTEMBER 1975



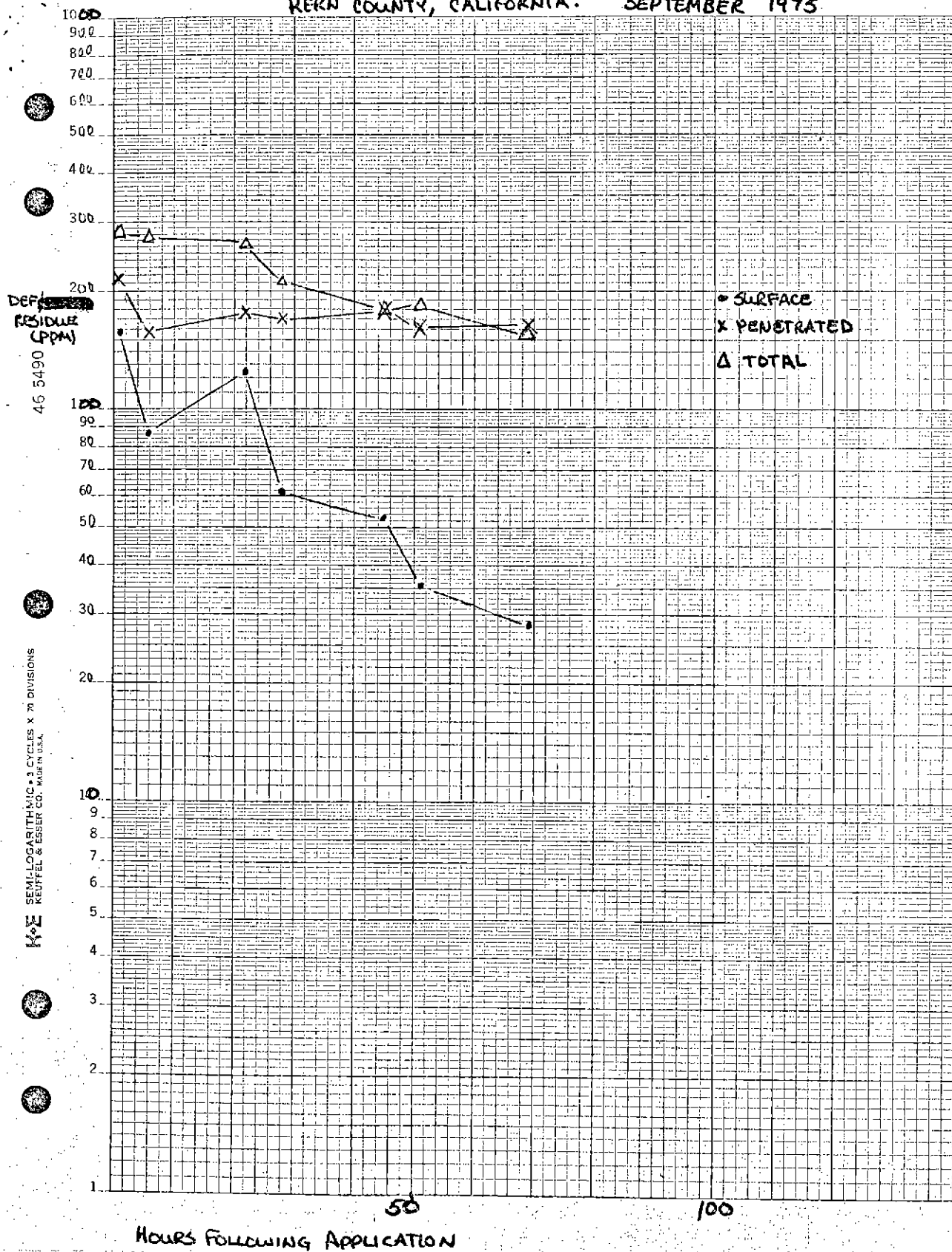
GRAPH 3: DEF ~~RESIDUES~~ RESIDUES ON COTTON IN FIELD 3 (PPM)
KERN COUNTY, CALIFORNIA. SEPTEMBER 1975



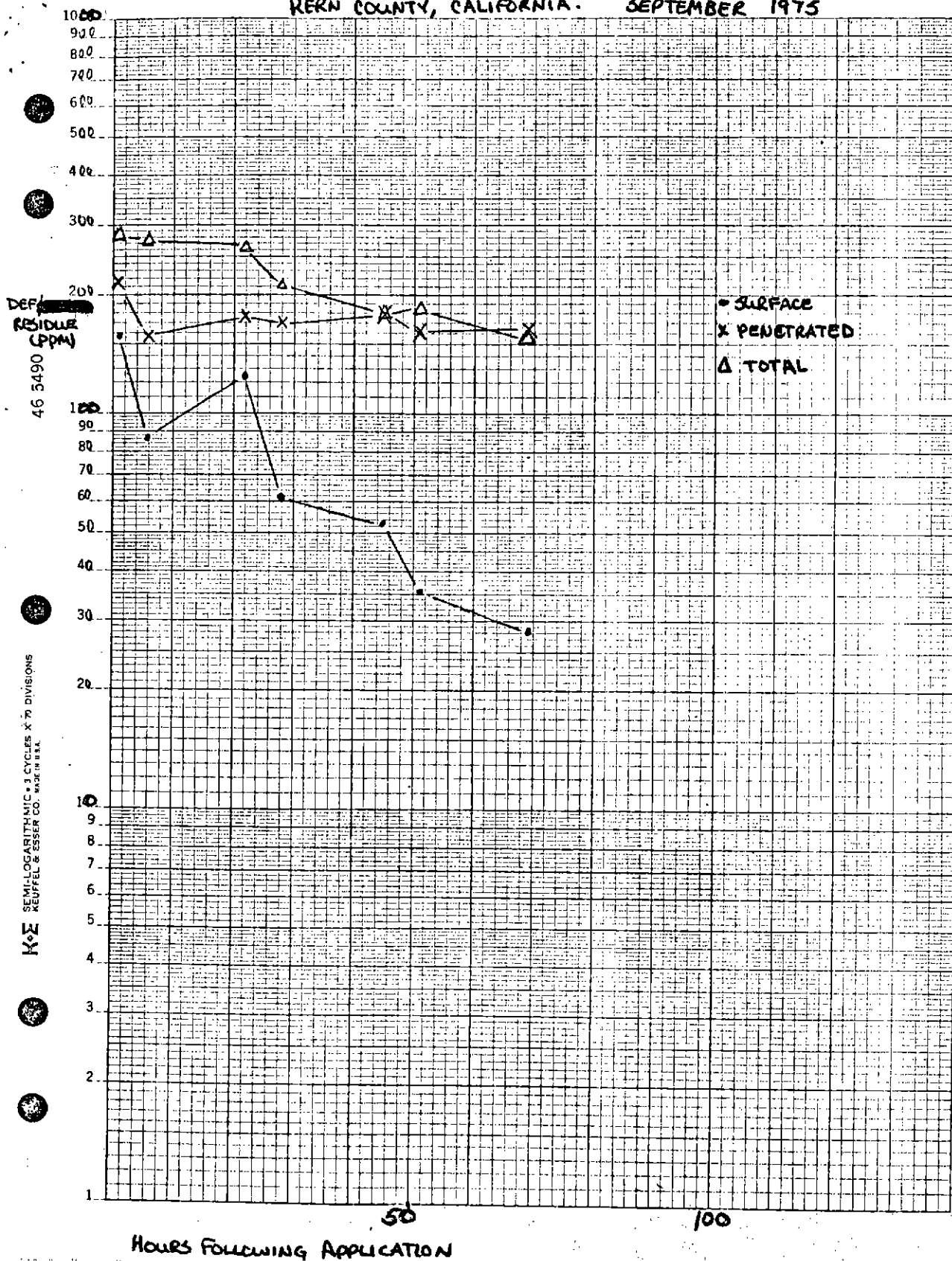
GRAPH 4: ~~FOLEX~~ FOLEX RESIDUE ON COTTON IN FIELD 4 (PPM)
KERN COUNTY, CALIFORNIA. SEPTEMBER 1975



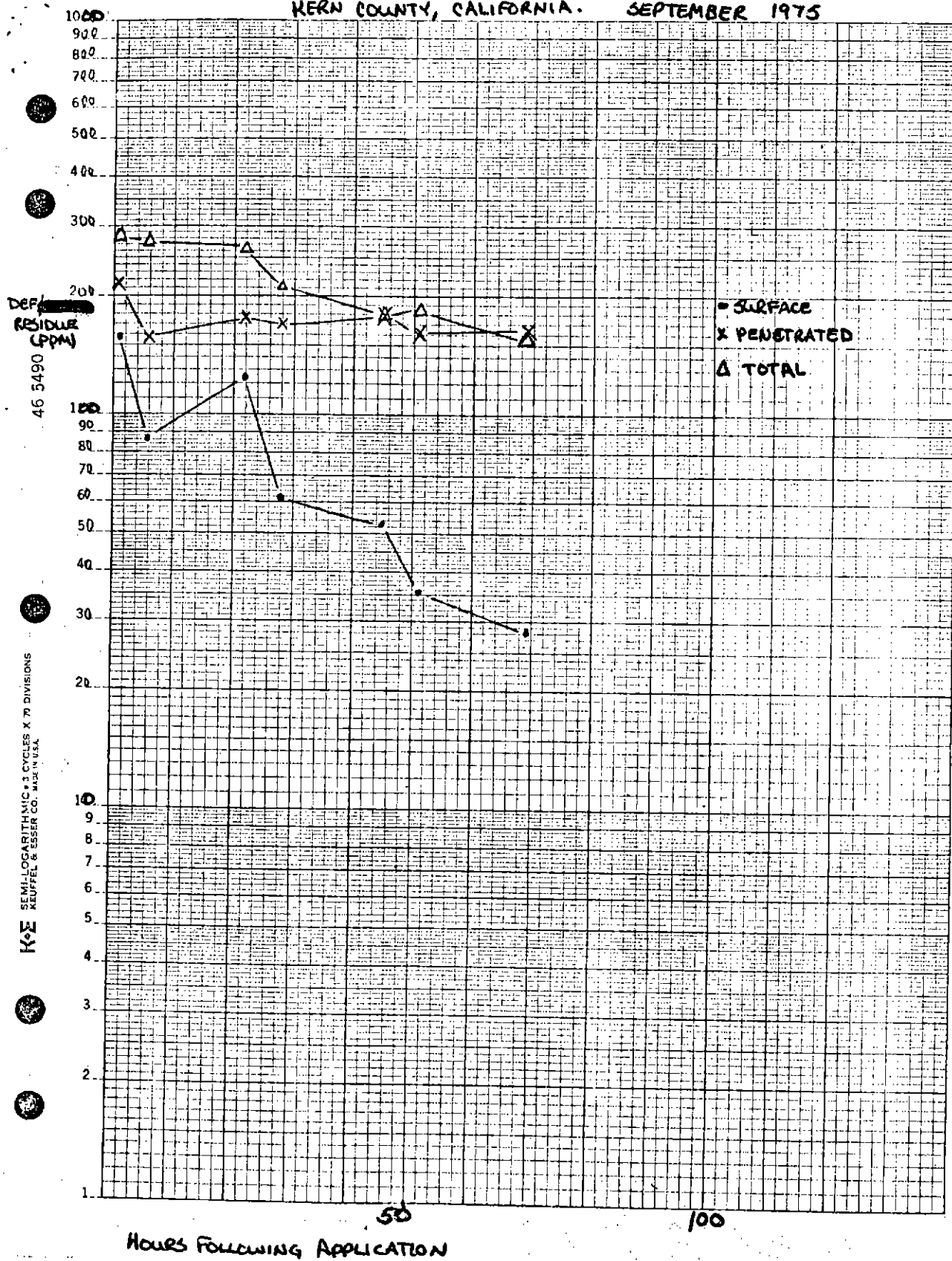
GRAPH 5: DEF ~~RESIDUE~~ RESIDUE ON COTTON IN FIELDS (PPM)
 KERN COUNTY, CALIFORNIA. SEPTEMBER 1975



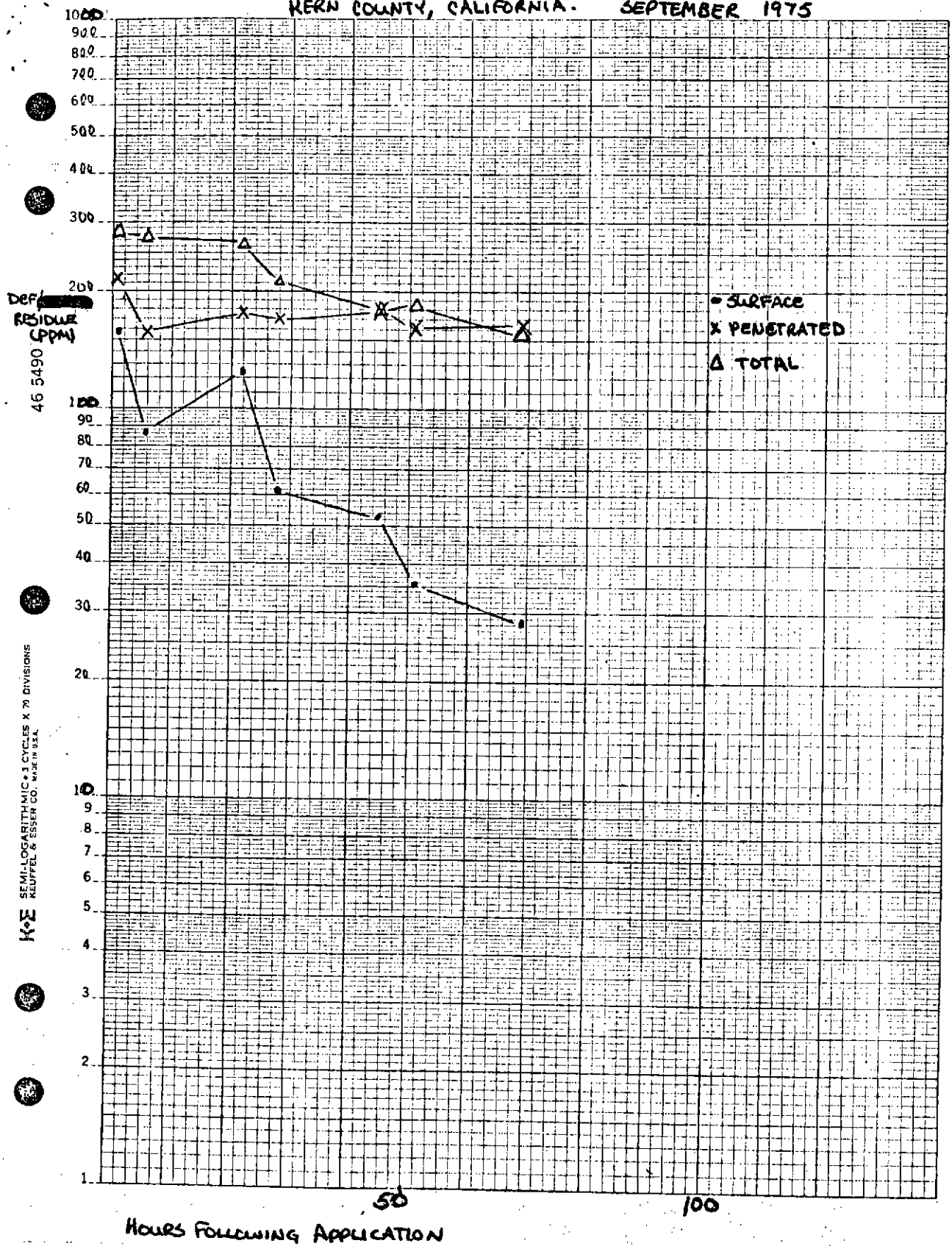
GRAPH 5: DEF ~~RESIDUE~~ RESIDUE ON COTTON IN FIELDS (PPM)
 KERN COUNTY, CALIFORNIA. SEPTEMBER 1975



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 KERN COUNTY, CALIFORNIA. SEPTEMBER 1975

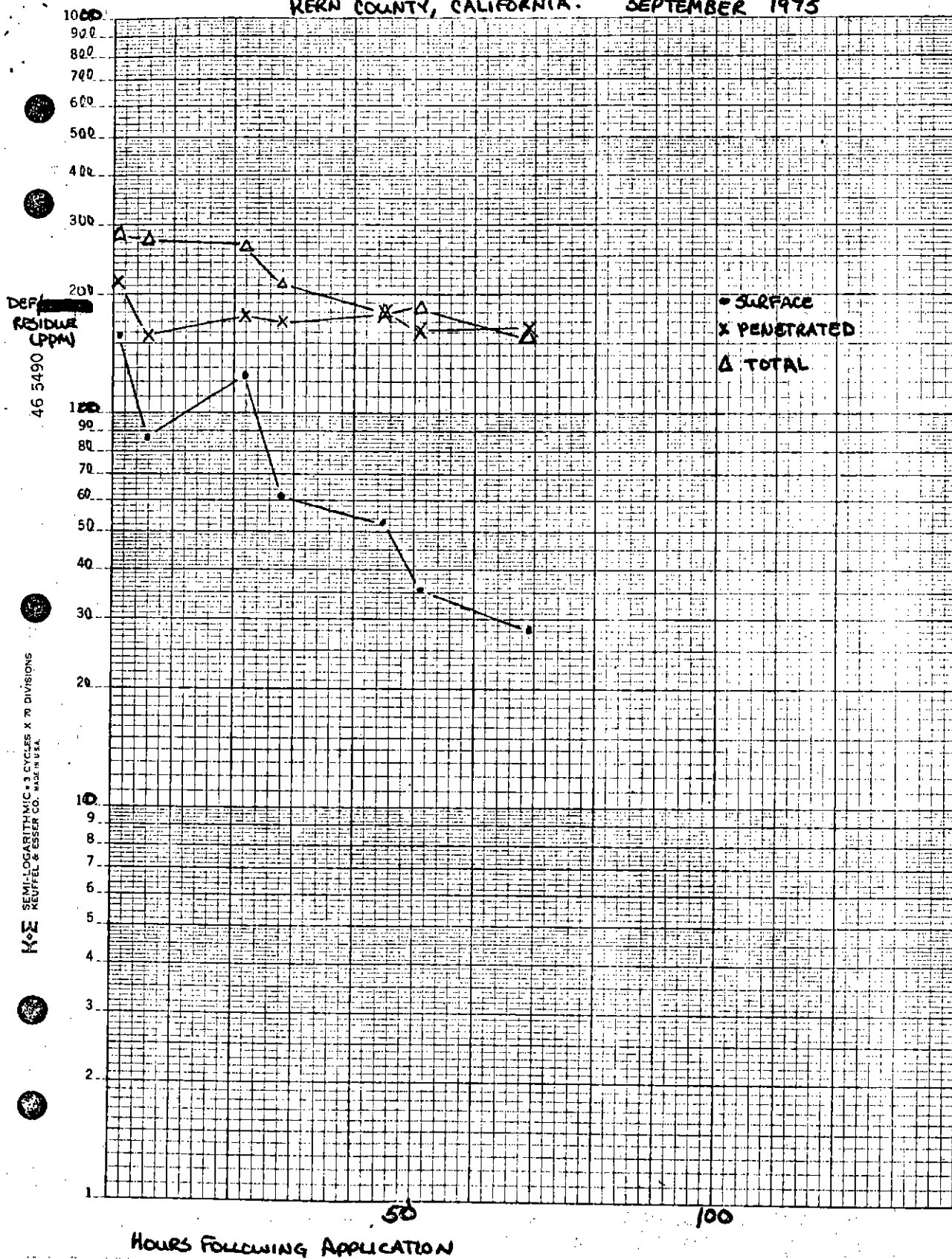


GRAPH 5: DEF [REDACTED] RESIDUE ON COTTON IN FIELD 5 (PPM)
 KERN COUNTY, CALIFORNIA. SEPTEMBER 1975



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GRAPH 5: DEF ~~RESIDUE~~ RESIDUE ON COTTON IN FIELDS (PPM)
 KERN COUNTY, CALIFORNIA. SEPTEMBER 1975



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 KEUFFEL & ESSER CO. MADE IN U.S.A.